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धूर्णी बम द्वारा संदमित खनिज रोधी तेल के आक्सीकरण के स्थायित्व की परीक्षण पद्धति

Indian Standard

OXIDATION STABILITY OF INHIBITED MINERAL INSULATING OIL BY ROTATING BOMB — METHOD OF TEST

UDC 621·315·615·2 : 543·872

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

November 1990

Price Group 3

Fluids for Electrotechnical Applications Sectional Committee, ETD 3

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 20 March 1990, after the draft finalized by the Fluids for Electrotechnical Applications Sectional Committee had been approved by the Electrotechnical Division Council.

This standard is considered of value in checking the oxidation stability of new mineral insulating oils containing 2, 6-ditertiary-butyl para-cresol in order to control the continuity of this property from shipment to shipment.

In preparing this standard, considerable assistance has been derived from ASTM D 2112-1986 'Standard test method for oxidation stability of inhibited mineral insulating oil by rotating bomb', issued by the American Society for Testing and Materials (ASTM).

In reporting the result of a test in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

OXIDATION STABILITY OF INHIBITED MINERAL INSULATING OIL BY ROTATING BOMB — METHOD OF TEST

1 SCOPE

1.1 This standard covers the method for the evaluation of the oxidation stability of new mineral insulating oils containing 2, 6-ditertiary-butyl para-cresol.

2 SUMMARY OF METHOD

2.1 The sample is agitated by rotating axially at 100 rev/min at an angle of 30° from the horizontal under an initial oxygen pressure of 6·33 kgf/cm^2 in a copper bomb (for rapid temperature equilibrium) with a glass sample container and copper catalyst coil, in the presence of water, at a bath temperature of 140°C. The time for an oil to react with a given volume of oxygen is measured; completion of the test is indicated by a specific drop in pressure.

3 APPARATUS

3.1 Oxidation Bomb

Glass sample container with cover and catalyst coil, pressure gauge thermometer, test bath, and accessories as described in Annex A, shall be used. The design of the apparatus is shown schematically in Fig. 1.

4 REAGENTS AND MATERIALS

4.1 Purity of Reagents

Reagent grade chemicals shall be used in all tests. Unless otherwise specified, it is intended that all reagents shall conform to the relevant Indian Standards where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is sufficiently high purity to permit its use without lessening the accuracy of the determination.

4.2 Emery Cloth, No. 00

4.3 Ethyl Ether

4.4 Isopropyl Alcohol, 99 Percent, Refined

4.5 Liquid Detergent

4.6 Oxygen, 99·5 Percent, with Pressure Regulation to 6.33 kgf/cm^2

4.7 Potassium Hydroxide, Alcohol Solution (1 Percent)

Dissolve 12 g of potassium hydroxide (KOH) pellets in 1 litre of 99 percent refined isopropyl alcohol.

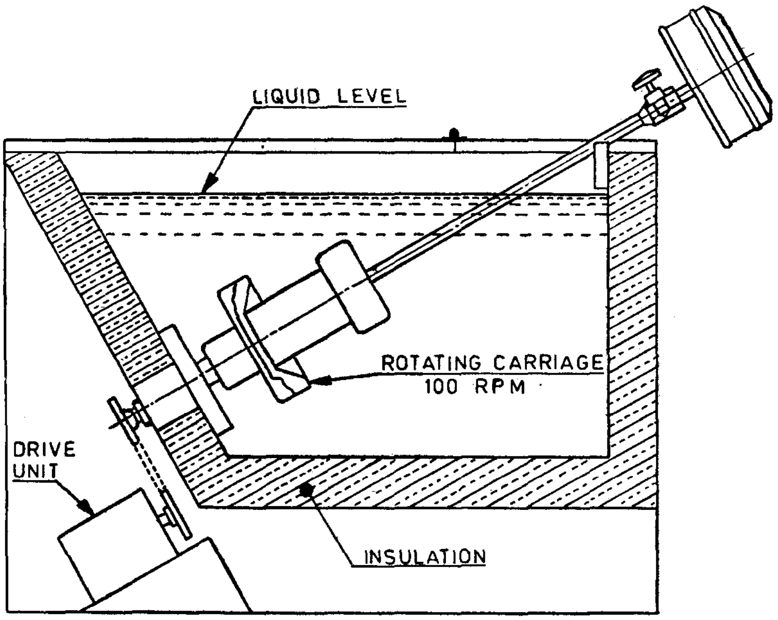


FIG. 1 ROTATING BOMB OXIDATION TEST APPARATUS, SCHEMATING DRAWING

4.8 Silicone Stopcock Grease

4.9 Wire Catalyst

1·628 mm diameter electrolytic copper wire.

5 PREPARATION OF APPARATUS

5.1 Catalyst Preparation

Immediately before use polish the copper wire with No. 00 emery cloth or its equivalent and wipe it with a clean dry cloth to make it free from abrasive. Wind 3 m of the wire into a coil having an outside diameter of 46 mm and stretch to a height of 40 mm. Clean the coil thoroughly with ethyl ether, air-dry, and insert immediately, with a twisting motion, inside the glass sample container and weigh to the nearest 0·1 g and record the weight.

5.2 Cleaning of Bomb

Wash the bomb body, lid, and inside of bomb stem with hot detergent solution and with water. Rinse inside of stem with isopropyl alcohol and blow-dry with clean dry air. If the bomb body lid, or inside of stem smells sour after simple cleaning, wash with alcoholic KOH solution and repeat as before.

NOTE — Insufficient cleaning of the bomb may adversely affect test results.

6 PROCEDURE

6.1 Charging

Weigh 50 ± 0·5 g of oil sample into the container, add 5 ml of distilled water, and cover with a 51 mm watch-glass. If rinsed water is present in the container, compensate for it by using less added water. Add 5 ml of distilled water to the bomb and slide the sample container and cover lid into the bomb body (*see* Note 1). Apply a thin coating of silicone stopcock grease to the O-ring bomb seal located in the gasket groove of the bomb lid to provide lubrication, and insert the lid into the bomb body. Place the bomb cap over the bomb stem; and tighten by hand. Cover the threads of the gauge-nipple with a thin coating of stopcock grease and screw the gauge into the top-centre tap of the bomb stem. Flush the bomb twice with oxygen supplied to the bomb at 6·33 kgf/cm^2 and release to the atmosphere. Adjust the regulating valve on the oxygen supply tank to 6·33 kgf/cm^2 at a room temperature of 25°C. For each 2·8°C above or below this temperature, add or substract 0·070 kgf/cm^2 unit to attain the required initial pressure. Fill the bomb to this required pressure and close the inlet valve securely by hand. If required, test the bomb for leaks by immersion in water (*see* Note 2). Prepare a duplicate sample in exactly the same way.

NOTES

1 The water between the bomb well and the sample container aids heat transfer.

2 If the bomb was immersed in water to check for leaks, dry the outside of the wet bomb by any convenient means such as an air blast or a towel. Such drying is advisable to prevent subsequent introduction of free water into the hot oil bath which would cause sputtering.

6.2 Oxidation

Bring the heating bath to the test temperature of 140°C while the stirrer is in operation. Insert the bombs into the rotating carriages and note the time. If an auxillary heater is used, keep it on for the first 5 minutes of the run and then turn it off (*see* Note 1). Allow the bath temperature to level out at the test temperature. This must occur within 10 minutes after the bombs are inserted. Maintain the test temperature within ± 0·1°C (*see* Note 2).

NOTES

1 The time for the bath to reach the operating temperature after insertion of the bombs may differ for different apparatus assemblies and should be observed for each unit. The objective is to find a set of conditions that do not permit a drop of more than 2°C after insertion of the bombs and allows the bomb pressure to reach a plateau within 15 minutes shown in curve A of Fig. 2.

2 Maintaining the correct temperature within the specified limits of ± 0·1°C during the entire test run is the most important single factor assuring good repeatability and reproducibility of test results.

6.3 Keep the bombs completely submerged and maintain rotation continuously and uniformly throughout the test. A standard rotational speed of 100 ± 5 rev/min is required. Any appreciable variations in this speed could cause erratic results. If a dial gauge is used, take readings every 5 minutes.

6.4 The test is complete after the pressure drops more than 1·76 kgf/cm^2 below the maximum pressure. The 1·76 kgf/cm^2 pressure drop usually, but not always, coincides with an induction-type 'period of rapid pressure drop'. When it does not, the operator should question whether he has produced a valid experiment.

NOTE — A valid experiment is shown as curve A in Fig. 2. The maximum pressure is reached in 10 to 15 minutes, a pressure plateau is established and an induction-type pressure drop is evidenced. Curve B, in which there is a gradual decrease in pressure before the induction break is recorded, is more difficult to evaluate since there is a question as to whether the pressure drop should be taken from the plateau or from the pressure at the induction break; usually, this type of curve derives from an improper experiment and should be considered suspect. Other cases (not illustrated), where the pressure continues to rise after the 10 to 15 minutes specified warm-up period, or where the pressure falls continuously from the maximum of the warm-up period, are certainly invalid experiments and should be repeated.

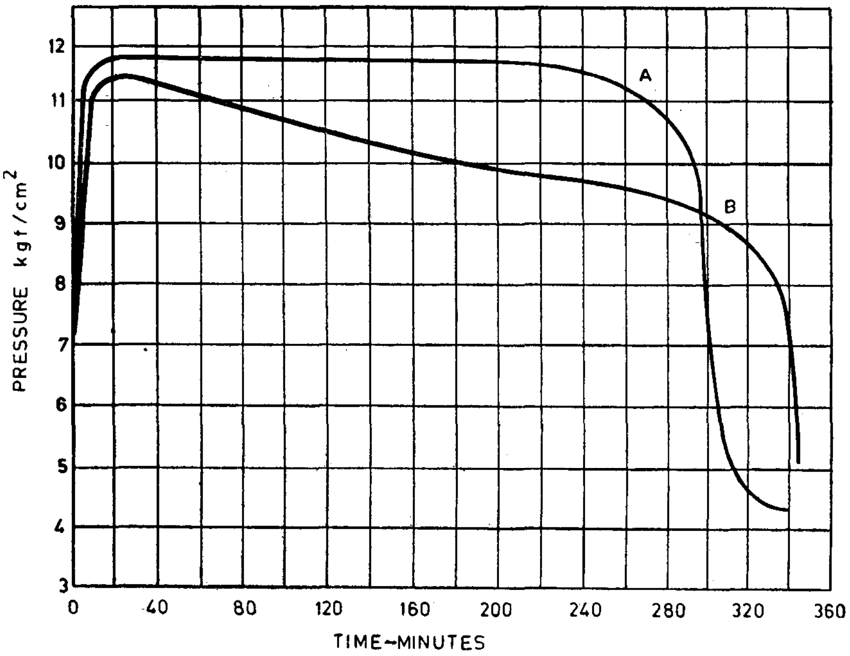


FIG. 2 CURVES OF TYPICAL TEST RUNS

6.5 After termination of the test, remove the bomb from the oil bath, dip briefly into and swirl around in a bath of light mineral oil to wash off the adhering bath oil. Rinse off the bomb with hot water, then immerse in cold water to bring them quickly to room temperature. Bleed off the excess oxygen pressure and open the bomb. This entire operation shall be completed within 3 minutes of the time the bomb is removed from the hot oil bath.

7 REPORT

7.1 Observe a plot of the recorded pressure versus time and establish the plateau pressure (*see* Note above). Also record the time at the point on the falling part of the curve where the pressure is 1·76 kgf/cm^2 less than the established plateau pressure. Plateau pressures in duplicate tests should not differ by more than 0·35 kgf/cm^2.

7.2 The bomb life of the sample is the time in minutes from the start of the test to a 1·76 kgf/cm^2 pressure drop from the level of the established plateau.

8 PRECISION

8.1 Establish repeatability and reproducibility by conducting experiments in different laboratories.

ANNEX A

(*Clause* 3.1)

ROTATING BOMB OXIDATION TEST APPARATUS

A-1 OXIDATION BOMB

A-1.1 The oxidation bomb, with lid, cap and stem shall be constructed as shown in Fig. 3.

A-1.2 The bomb body and lid shall be machined from 76 mm solid copper rod for maximum rate of heat transfer. The interior surface shall be given a smooth finish to facilitate cleaning. The bomb body and lid shall be heavily chrome-plated.

A-1.3 The bomb stem shall be made of stainless steel, having an inside diameter of 6·35 mm and shall be equipped with a 6·35 mm needle valve.

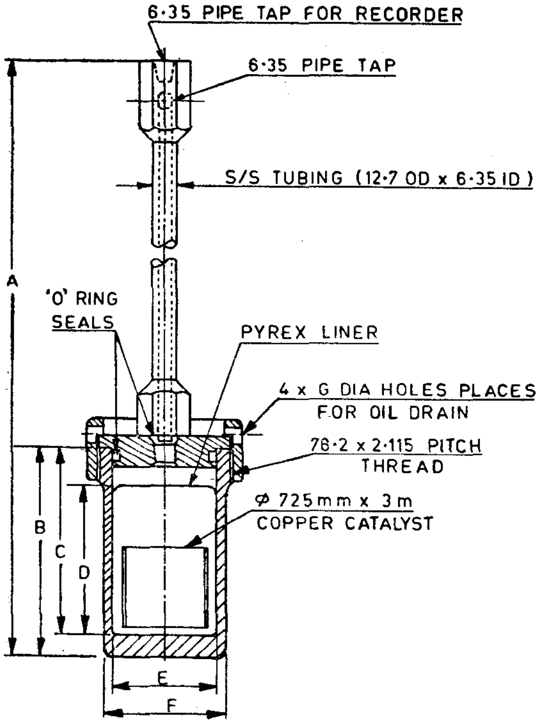
A-1.4 The bomb cap (or closure ring) shall be made of plated steel.

A-1.5 The bomb shall withstand a working pressure of 35 kgf/cm^2 at 150°C.

A-1.6 O-ring Gaskets — TFE-fluorocarbon resin reinforced silicone, 50·8 mm in inside diameter by 60·3 mm in outside diameter shall be used.

A-2 GLASS SAMPLE CONTAINER

A-2.1 The glass sample container, 175-ml capacity, with copper catalyst coil, shall be constructed of borosilicate glass as shown in Fig. 4.


6·35 PIPE TAP FOR RECORDER
6·35 PIPE TAP
S/S TUBING (12·7 OD x 6·35 ID)
A
'O' RING SEALS
PYREX LINER
4 x G DIA HOLES PLACES FOR OIL DRAIN
76·2 x 2·115 PITCH THREAD
φ 725 mm x 3 m COPPER CATALYST
B
C
D
E
F

All dimensions in millimetres.

Material

Cap, steel
Body, copper } hard chrome plated
Lid, copper
Stem, S/S

	mm
A	536·58
B	120·65
C	107·95
D	86 to 89
E	60·325/60·579
F	69·85
G	9·525

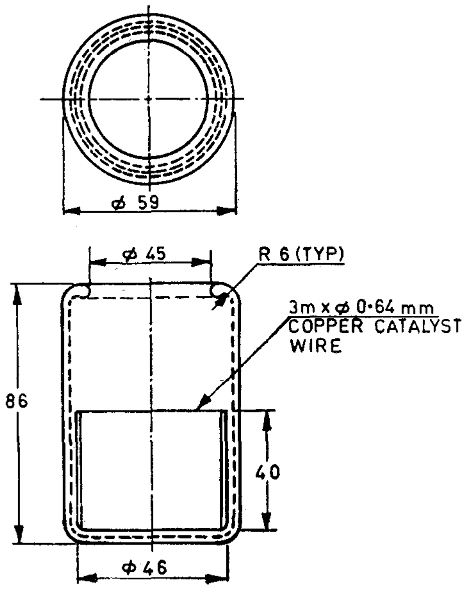
FIG. 3 CONSTRUCTION OF OXIDATION BOMB

A-2.2 The top of the sample container shall be covered with 50·8 mm diameter watch glass. The watch glass shall have fire polished edges.

A-2.3 The glass sample container shall have a sliding fit in the bomb with no excess side clearance. The container alone shall have a maximum wall thickness of 2·5 mm and shall not weigh more than 100 g.

A-3 GAUGE

A-3.1 The gauge, recording (*see* Fig. 5) or indicating, shall have a range from 0 to 14 kgf/cm² and shall be graduated in 0·4 kgf/cm² divisions.



All dimensions in millimetres.

FIG. 4 GLASS SAMPLE CONTAINER WITH CATALYST

A-3.2 The accuracy shall be 3 percent or less of the total scale interval.

A-3.3 Recording gauges shall be mounted so that the face is perpendicular to the axis of rotation.

A-4 OXIDATION BATH

A-4.1 The oxidation bath shall be equipped with an efficient stirrer and a suitable device for holding and rotating the bomb axially at an angle of 30 degrees at 100 ± 5 rev/min while submerged in oil to a point at least 25·4 mm below of the bath liquid.

A-4.2 A bath at least 230 mm deep, filled with 30·3 litres of heavy bath oil per bomb, has the proper heat capacity. Metal block baths are not satisfactory for this service.

A-4.3 Thermal regulation shall be provided to maintain the bath within ± 0·1°C of the test temperature (140°C) for periods as long as 8 hours; there should be sufficient heat available to bring the bombs to operating temperature within 10 to 15 minutes.

A-5 THERMOMETER

A-5.1 Thermometer having a range from 120 to 150°C, graduated in 0·1°C intervals. Place the thermometer in the bath so that it is submerged to the immersion mark.

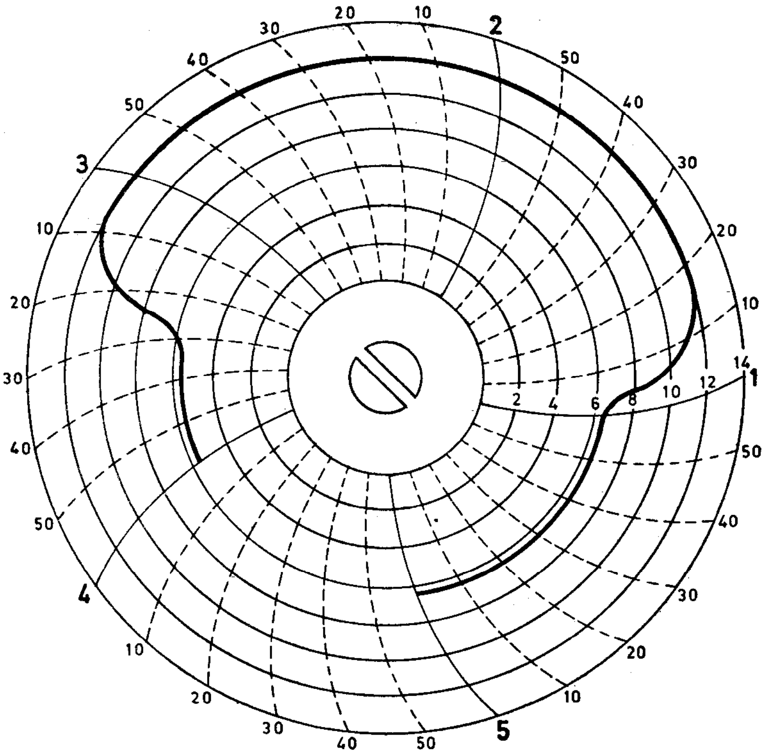


FIG. 5 CHART OF RECORDING PRESSURE GAUGE (ACTUAL SIZE = 114 mm)

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